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INTERLABORATORY STUDY 89-1

VOLATILE ORGANIC PARAMETERS IN
REAGENT WATER AND OIL AND GREASE
IN REAGENT WATER AND EFFLUENT

JULY 1990





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JULY 1990



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1 SUMMARY AND CONCLUSIONS

Interlaboratory Study 89-1 was the second of an on-going program of laboratory performance management studies initiated by the Quality Assurance Office, Laboratory Services Branch of the Ontario Ministry of the Environment. It assesses the analytical variability of selected organic parameters (see Sections 3.1.2 and 3.1.3) in spiked reagent water and Oil and Grease (Solvent Extractables) from reagent water and one effluent sample. Sixteen laboratories initially agreed to participate in the study. Results for the Volatile Organic samples were reported from ten participants. Results for the Oil and Grease samples were reported from twelve of the participants.

The results demonstrate variable performance between laboratories. Some participants were able to achieve good recovery (80-110%) of the target parameters across the scan. Other laboratories demonstrated biases due to differences in calibration standards or analytical procedures.

Differences in standards may result in consistent over- or under-recovery across the scan. Differences in analytical procedures may produce a variety of effects, including over- or under-recovery of specific parameters, patterns of increasing or decreasing recovery across the scan, and variability of recovery of low level spikes versus high level spikes. It is desirable that laboratories demonstrating some of these problems strive to correct these difficulties. Future interlaboratory studies that include the analysis of Volatile Organics will attempt to monitor improvements in performance by the participants.

Due to the large number of parameters included in the Volatile scan, this report does not attempt to assess the between-laboratory performance of individual parameters. A review of the range of results given by the Minimum and Maximum values in Table 1, Appendix 1, indicates that the results may vary by an order of magnitude. However, as there were only two spiked samples submitted to each participant, there is insufficient data to assess the analytical characteristics of each parameter. Future studies will attempt to assess the analytical characteristics of individual parameters by submitting more samples to the participants and including duplicate analyses.

The results for the Oil and Grease samples demonstrate that, regardless of solvent used, individual laboratories demonstrate systematic bias. The participants demonstrated good within-laboratory precision, but the between-laboratory variability had a significant effect on the data distribution. The analytical techniques used to perform this test may affect the variability of performance (3) between the participants. Future studies will attempt to monitor improvements in performance for this analytical method.

The following table summarizes the individual laboratory's performance.

TABLE I - SUMMARY OF PARTICIPANTS' PERFORMANCE

LAB CODE	MISA TEST GROUP	PERFORMANCE
1001	Volatiles (16 & 17)	- Results from only one sample (second sample bro- ken in transit), therefore not possible to assess within-laboratory precision
		- Variable performance across scan
		 Reported MDL's for Bromomethane, Chloromethane and Vinyl chloride greater than specified in MISA General Regulation, therefore not possible to determine if there was background contamination for these parameters
	Oil and Grease (25)	- Excellent recovery relative to design values but appear high relative to other participants
		- Possible background laboratory contamination
1002	Volatiles (16 & 17)	- Good recovery relative to design values
		- Consistent performance across scan except for 5 specific parameters
		- Some laboratory contamination with Dichlorome- thane, but at a very low level
	Oil and Grease (25)	- Excellent recovery relative to design values but appear high relative to other participants
		- No evidence of laboratory background contamination
1003	Volatiles (16 & 17)	- Good recovery relative to design values
		- Consistent performance across scan except for 5 specific parameters
		- No evidence of laboratory background contamination
	Oil and Grease (25)	- Variable performance: good recovery of the low spike but under-recovered the high spike
		- No evidence of laboratory background contamination

LAB CODE	MISA TEST GROUP	PERFORMANCE
1004	Volatiles (16 & 17) Oil and Grease (25)	 No results reported for these samples Good recovery relative to the design values Good agreement with the interlaboratory mean and median No evidence of laboratory background contamination Very high result for the effluent sample; result was deleted from statistical calculations
1005	Volatiles (16 & 17)	 Variable within-laboratory precision Good recovery of low spike for most parameters but many early-eluting parameters under-recovered in high spike Consistent pattern of recovery across scan for low spike but pattern of increasing recovery across scan for high spike No evidence of laboratory background contamination
1007	Oil and Grease (25) Volatiles (16 & 17)	 No results reported for these samples Biased low relative to design values and interlabo-
***************************************	· outres (10 to 17)	ratory mean - Very low background level of Dichlorobenzes but no other evidence of background contamination - Pattern of increasing recovery across the scan in low spike but no pattern demonstrated in high spike
	Oil and Grease (25)	 Excellent recovery relative to design values but appear high relative to other participants Good within-laboratory precision No evidence of laboratory background contamination

Some laboratory contamination with Toluene, but at

- Good recovery of the low spike relative to the design value but under-recovered the high spike

Low spike result was in good agreement with the interlaboratory mean and median, but biased low for the high spike
 No evidence of laboratory background contamina-

LAB CODE	MISA TEST GROUP	PERFORMANCE	
1008	Volatiles (16 & 17) Oil and Grease (25)	 No results reported for these samples Biased low relative to the design values and the interlaboratory mean 	
		 Good recovery relative to the interlaboratory median Good within-laboratory precision No evidence of laboratory background contamination 	
1009	Volatiles (16 & 17)	- Good recovery relative to the design values for the low spike but biased low for the high spike	
		- Variable within-laboratory precision	

tion

Oil and Grease (25)

a very low level

LAB CODE	MISA TEST GROUP	PERFORMANCE
1010	Volatiles (16 & 17)	 Variable performance relative to the design values Pattern of recovery across the scan similar for both spiked samples Laboratory contamination with Dichloromethane
	Oil and Grease (25)	 Biased low relative to the design values and the interlaboratory mean and median Consistent within-laboratory performance Some possible laboratory background contamination
1011	Volatiles (16 & 17) Oil and Grease (25)	 Did not participate in this portion of the study Good recovery of the high spike relative to the design value but under-recovered the low spike High spike result was in good agreement with the interlaboratory mean and median, but biased low for the low spike No evidence of laboratory background contamination
1012	Volatiles (16 & 17) Oil and Grease (25)	 Variable recovery relative to the design values Similar pattern of recovery across the scan demonstrated in both spiked samples Unusual laboratory contamination with Chloromethane Laboratory contamination with Dichloromethane No results reported for these samples

AB CODE	MISA TEST GROUP	PERFORMANCE
)13	Volatiles (16 & 17)	- Good recovery relative to design values
		- Consistent performance across scan except for 3 specific parameters
		- No evidence of laboratory background contamination
	Oil and Grease (25)	- Biased low relative to the design values and to the inter-laboratory mean and median
		- Variable within-laboratory precision
		- No evidence of laboratory background contamination
14	Volatiles (16 & 17)	- Variable recovery relative to the design values
		- Laboratory contamination with Dichloromethane
		 Recovery across scan clusters into three groups; demonstrated in both spiked samples
	Oil and Grease (25)	- Biased low relative to the design values
		- Good recovery relative to the interlaboratory mean and median
		- Good within-laboratory precision
		- No evidence of laboratory background contamination
15	Volatiles (16 & 17)	- No results reported for these samples
	Oil and Grease (25)	- Good recovery relative to the design values
		- Good recovery relative to the interlaboratory mean and median
		- Good within-laboratory precision
		- No evidence of laboratory background contamination

2 INTRODUCTION

Interlaboratory performance studies, or round robins, are conducted to assess the comparability of data among different laboratories. These studies help in the identification of biases and precision or accuracy problems. Participation in such studies helps in improving individual laboratory performance and maintaining performance standards. The Quality Assurance Office, Laboratory Services Branch (LSB) of Environment Ontario has instituted an on-going program of interlaboratory studies to assess and enhance the performance of environmental laboratories providing analytical services.

Interlaboratory Study 89-1 consists of two components. Part 1 was designed to assess the analytical variability of volatile organic parameters in fortified reagent water. Part 2 was designed to assess the analytical variability of Oil and Grease (solvent extractables) from fortified reagent water and an effluent sample. The parameter list was chosen from analytical test groups listed in the MISA (Municipal and Industrial Strategy for Abatement) General Regulation (1). Participants were requested to use methods which conformed to the MISA analytical principles and protocols given in the General Regulation (1).

Sixteen laboratories agreed to participate in this interlaboratory study. A list of participants is included in Appendix 2. Not all of the participants produced final results for all of the samples submitted (see Section 3.4). Final percent participation was as follows: Volatiles - 69%, Oil and Grease - 75%.

Two sets of samples (7 in total) were distributed to each of the 16 participants. Each set consisted of three samples for volatile organic analysis (MISA Test Groups 16 and 17), and four samples for Oil and Grease analysis (MISA Test Group 25). Details of sample preparation and distribution are given in Sections 3.1 and 3.2. Analytical methodology and data handling are presented in Sections 3.3 and 3.4. Final results are presented and laboratory performance is discussed in Section 4.0.

3 PROCEDURE

3.1 Preparation of Samples

3.1.1 Preparation of Matrices

a) Reagent Water

Volatile Samples

Four litres of deionized, distilled water was collected in a clean Erlenmeyer flask. It was purged with a gentle stream of nitrogen gas for approximately two hours. This process was carried out in a separate laboratory, well removed from routine organic laboratory atmosphere to avoid contributing artifacts to the analysis. Any contact of water with plastic materials was avoided.

Oil and Grease Samples

Non-purged, deionized, distilled water was used for the Oil and Grease samples. Care was taken that no plastic materials came into contact with the water.

b) Effluent Matrix

A bulk effluent sample was collected from extra samples received at Laboratory Services Branch. The effluent was stirred overnight to form a homogeneous mixture using a stirrer with a Teflon shaft and propeller.

3.1.2 Preparation of Volatile Samples

A combined spiking solution was prepared in purged, distilled, deionized water. Stock solutions consisted of three different volatile organic mixtures purchased from Supelco, and one in-house prepared ampoule. Table II lists the mixtures of organic parameters stated to be present in the ampoules by the supplier.

TABLE II - VOLATILE ORGANICS PARAMETER LIST

Purgeable A Carbon Tetrachloride

Chlorobenzene 2-Chlorovinyl ether

Chloroform
Dibromochloromethane
1,1,-Dichloroethane

1,1-Dichloroethylene 1,2-Dichloropropane

Methylene chloride (Dichloromethane)

Tetrachloroethylene 1,1,2-Trichloroethane Trichloroethylene Trichlorofluoromethane

Purgeable B Benzene

Bromodichloromethane

Bromoform
1.2-Dichloroethane

t-1,2-Dichloroethylene

1,3-Dichloropropene (both isomers)

Ethyl benzene

1,1,2,2-Tetrachloroethane

Toluene

1,1,1-Trichloroethane

Purgeable C Bromomethane

Chloroethane Chloromethane Vinyl chloride

p-Xylene

VOLA02

(In-house ampoule) m-Xylene o-Xylene

1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Ethylene dibromide

Samples for volatile analysis were prepared in 40 mL glass vials that had screw caps with Teflon-lined septa. Sample vials were filled almost to overflowing with the sample matrix, with the miniscus rising above the edge of the vial. The volume of 40 mL was confirmed by weighing the vials before and after filling (± 1 mL). Unspiked samples were labelled "VOL A".

All sample spiking was done using 50 or 500 microlitre syringes, depending on the spiking level. The spiking solution was expelled from the syringe below the surface of the matrix. The screw cap septa (Teflon side towards the sample) was carefully slid across the top of the

vial and quickly screwed on, so as to avoid trapping any air bubbles in the vial. The samples were gently inverted three or four times to mix the contents. The low spike samples were labelled "VOL B" and the high spike samples were labelled "VOL C".

3.1.3 Preparation of Oil and Grease Samples

Samples for Oil and Grease analysis were prepared using amber 1000 mL glass bottles. To each sample bottle, 1000 mL of distilled, deionized water was added by weight (1000 g ± 2 g). It was assumed that the density of the water equalled 1.0. The unspiked samples were labelled "O&G A".

A spiking solution was prepared by weighing an appropriate amount of cooking oil in a volumetric flask, and diluting to volume with acetone. Spiking was done using 0.5 and 1.0 mL gas-tight syringes. The low spike was labelled "O&G B" and the high spike was labelled "O&G C".

The homogenized bulk effluent was added to each sample bottle by weight (1000 g ± 2 g). Any possible error introduced by the density of the effluent was considered too small to be significant. These samples were not spiked with the solution used for the reagent water samples. They were labelled as "O&G D".

3.2 Sample Distribution

Prior to sample preparation a letter of notification was sent to each participating laboratory. Confirmation of participation was received by letter or telephone. A list of participating laboratories and examples of correspondence is given in Appendix 2.

The two sets of three and four samples (Volatiles and Oil and Grease respectively) for each participating laboratory were packaged in cardboard boxes and shipped via Purolator courier on January 31, 1989. One laboratory reported that one cap came off one sample bottle for Oil and Grease during transit. Another laboratory reported that one sample for Volatiles was broken in transit. A third laboratory reported that they did not receive the package containing the Volatile samples. These samples were not replaced.

3.3 Analytical Methodology

Participants were requested to analyze the samples using routine in-house methods that complied with the principles and protocols outlined in Schedule 3 of the MISA General Regulation (1).

Participants were not required to provide detailed information regarding instrumental methodology. Some participants provided information regarding instrument model and column used, and all participants stated that additional information was available on request. Participants were

asked to indicate which solvent was used for the Oil and Grease analyses. Participants were subsequently contacted and requested to indicated which type of gas chromatograph column had been used for the analysis of the Volatile Organics.

3.4 Data Handling

Results were submitted to the Quality Assurance Office, LSB in written form by mail. All data were manually entered by laboratory code into an electronic spreadsheet. Blank spaces were left when a laboratory did not report results for a specific parameter that was present in the spiking material. A "0" was entered when a laboratory reported values for a specific parameter in some samples, but reported "Not Detected" in other spiked samples.

Final percent participation was as follows: Volatiles - 69%, Oil and Grease - 75%.

Interlaboratory variability was determined by calculating the mean, median, and standard deviation from the results reported (n=11 for Volatiles). The minimum and maximum values were identified to give an indication of the range. Outliers were not removed from the data set when calculating interlaboratory variability as the range of results precluded a selection of participants demonstrating a central tendency.

The results for the Oil and Grease were divided into two groups, depending on the solvent used for the extraction by the participants. The mean, median, minimum, maximum, and standard deviations were calculated for each group (n=7 for Freon and n=5 for Dichloromethane). Sample O&G A (Blank Reagent Water), had several results reported "< value". To perform the calculations, one half of the reported "value" was used. One result for O&G D was deleted when calculating the mean and standard deviation from the participants using Dichloromethane.

Results were converted to percent recovery based on the design value of the spiking material for both data sets. These values are presented in Tables 2 and 4 in Appendix 1. The results were not blank corrected.

Appendix 1 includes bar graphs of the recovery for each parameter from each participating laboratory (Figures 1-6 for Volatiles and Figures 7-8 for Oil and Grease). For the Volatiles, parameters are arranged left to right in order of gas chromatographic elution (based on a SPB-5 capillary column). Outliers were not deleted from the data set when preparing the graphs. All results are presented as percent recovery relative to the design value. Different participants reported the results for o-, m-, and p-Xylenes in varying combinations. The graphs have been separated as follows:

- (i) Figure 1 has two laboratories reporting the three isomers separately,
- Figures 2, 3, 5, and 6 have seven laboratories reporting m- and p-Xylenes together and o-Xylene separately, and
- (iii) Figure 4 has one participant reporting o- and p-Xylenes together and m-Xylene separately.

Figure 9 is a bar graph of the results from each participant for the bulk effluent sample (O&G D). Results are presented as mg/L and separated according to the two different solvents used.

4 RESULTS AND DISCUSSION

An overview of the interlaboratory performance for Volatiles is described. Individual performance of each participant is described separately and was assessed according to the following criteria: recovery of each parameter relative to the design values, laboratory background contamination of selected parameters, and consistency of performance within the scan (based on a SPB-5 capillary column).

On overview of the interlaboratory performance for Oil and Grease is described. Individual performance of each participant is described separately and was assessed according to the following criteria: recovery relative to the design values and performance relative to the interlaboratory mean and median.

4.1 Volatile Samples

OVERVIEW OF INTERLABORATORY PERFORMANCE - VOLATILES

The results demonstrate variable performance between laboratories. Some participants were able to achieve good recovery (80-110%) of the target parameters across the scan. Other laboratories demonstrated biases due to differences in calibration standards or analytical procedures.

Differences in standards may result in consistent over- or under-recovery across the scan. Differences in analytical procedures may produce a variety of effects, including over- or under-recovery of specific parameters, patterns of increasing or decreasing recovery across the scan, and variability of recovery of low level spikes versus high level spikes. It is desirable that laboratories demonstrating some of these problems strive to correct these difficulties. Future interlaboratory studies that include the analysis of Volatile Organics will attempt to monitor improvements in performance by the participants.

Due to the large number of parameters included in the Volatile scan, this report does not attempt to assess the between-laboratory performance of individual parameters. A review of the range of results given by the Minimum and Maximum values in Table 1, Appendix 1, indicates that the results may vary by an order of magnitude. However, as there were only two spiked samples submitted to each participant, there is insufficient data to assess the analytical characteristics of each parameter. Future studies will attempt to assess the analytical characteristics of individual parameters by submitting more samples to the participants and including duplicate analyses.

LABORATORY 1001

Sample VOL C (High Spike) was broken in transit and therefore results were not reported for this sample. This laboratory did not report results for Trichlorofluoromethane. They used a DB-1 60 m, 0.25 mm I.D. capillary gas chromatographic column.

Recovery of the parameters from Sample VOL B was variable, relative to the design values. Early eluting parameters were over-recovered, except for 1,1-Dichloroethylene. Late eluting parameters were under-recovered, particularly trans-1,3-Dichloropropylene, Tetrachloroethylene, Chlorobenzene, and 1,1,2,2-Tetrachloroethane.

Results for the blank sample (VOL A) were all reported as <MDL. For most parameters, the results suggest that there was no background contamination in this laboratory. For three parameters (Bromomethane, Chloromethane, and Vinyl chloride) the MDL was <10 $\mu g/L$. The MISA Regulation MDLs are 3.7, 3.7, and 4.0 respectively. It is difficult to determine whether there was any background contamination for these three parameters.

Performance across the scan was very variable. As results were reported from only one sample (VOL C was broken in transit), it is not possible to assess the consistency of performance of this laboratory.

LABORATORY 1002

This laboratory did not report any problems with the analysis of the samples. They used a DB-5 capillary gas chromatographic column.

The results demonstrated good recovery relative to the design values, for the majority of the parameters. Recovery varied between 85-115% for both the low and high spiked samples (VOL B and VOL C, respectively). There were some problem parameters that were under-recovered, particularly Trichlorofluoromethane, Carbon Tetrachloride, Tetrachloroethylene, and m-& p-Xylenes. Chloromethane and 1,2-Dichlorobenzene were over-recovered by a noticeable amount.

A small amount $(1.7 \,\mu\text{g/L})$ of Dichloromethane was reported in the blank sample (VOL A). This is a common laboratory contaminant, but does not appear to pose a problem. The results for Dichloromethane in the spiked samples were not corrected by this value.

Some parameters appear to present some problems for analysis by this participant. As mentioned above, there were several parameters that were under-recovered (<60% recovery). The patterns of recovery were the same in both the low and high spiked samples (Figures 2 and 5). These results suggest that this laboratory has consistent performance, but needs to address some specific problem parameters.

LABORATORY 1003

This laboratory did not report any problems with the analysis of the samples. They used a J & W DB-624 30 m, 0.032 I.D. capillary gas chromatographic column.

The results demonstrated good recovery relative to the design values, for many of the parameters. Recovery ranged between 80-110% for both the low and high spiked samples (VOL B and VOL C, respectively). 1,1,2-Trichloroethane and Ethylene Dibromide were over-recovered in both samples (140% and 136% in the low spike, and 168% and 149% in the high spike). Low recovery (<60%) was observed for Trichlorofluoromethane, 1,1-Dichloroethylene, and Carbon Tetrachloride in both samples.

Nothing was reported in the blank sample (VOL A), so there was no evidence of any laboratory contamination problems.

Similar patterns of recovery were observed for both the low and high spiked samples (Figures 2 and 5). The same parameters were over- or under-recovered in both samples, as noted above. These results suggest that this laboratory has consistent performance but needs to address some specific problem parameters, as noted above.

LABORATORY 1005

This laboratory did not report any problems with the analysis of the samples. They used a SE-30 m, 0.025 mm I.D. capillary gas chromatographic column.

The results for the low spike (Sample VOL B) demonstrate good recovery relative to the design values, for most of the parameters. Several parameters were under-recovered, particularly Trichlorofluoromethane, 1,1,-Dichloroethylene, Carbon Tetrachloride, and Tetrachloroethylene. The recovery (relative to the design values) was not consistent in the high spike (VOL C). Many parameters, particularly at the beginning of the scan, were under-recovered.

Nothing was reported in the blank sample (VOL A), so there is no evidence of any laboratory contamination problems.

The results across the scan for the low spike present a consistent pattern of recovery (Figure 2), except for the problem parameters noted above. However, for the high spike there is a pattern of increasing recovery (Figure 5), except for Tetrachlorocthylene. These results suggest that this laboratory has some difficulty analyzing samples of a higher concentration.

LABORATORY 1007

This laboratory did not report any problems with the analysis of these samples. They used a DB-5 30 m capillary gas chromatographic column.

The results from both spiked samples were under-recovered relative to the design values, except for o-Xylene in the low spike (VOL B). These results were also low relative to the interlaboratory mean for both samples. This suggests that their calibration standard differs from the spiking material used by the QA Office of MOE and also differs from the calibration standards of the other participants.

Values of less than 1.0 µg/L were reported for the three Dichlorobenzenes in the blank sample (VOL A). Values this low should not present any problems of laboratory contamination. No other evidence of laboratory contamination is apparent in the results.

The results from the low spike demonstrate a pattern of increasing recovery across the scan (Figure 3). The loss of the first seven parameters in the scan from this sample suggests that some adjustments are necessary to the temperature programming. This pattern is not evident in the results from the high spike (Sample VOL C, Figure 6), but there are several early eluting parameters that had very low recoveries (<20%). Some individual parameters in the middle of the scan also appear to be difficult for this laboratory to analyze, particularly Carbon Tetrachloride and trans-1,3-Dichloropropylene. There are several problem areas that need to be addressed by this laboratory.

LABORATORY 1009

This laboratory did not report any problems with the analysis of these samples. They used a Restek TRX-Volatiles 60 m, 0.032 mm I.D. capillary gas chromatographic column.

The results for the low spike (Sample VOL B) demonstrate good recovery relative to the design values, for most of the parameters. Four parameters were under-recovered (Trichlorofluoromethane, Dichloromethane, Carbon Tetrachloride, and Tetrachloroethylene). Bromomethane was over-recovered in both spiked samples. The results from the high spike (VOL C) were low, relative to the design value. The same parameters that had low recoveries for the low spike were also much lower for the high spike.

A value of $1.1~\mu g/L$ was reported for Toluene in the blank sample (VOL A). This is a common laboratory contaminant, but at this low level does not appear to pose a problem. The value reported in the blank for Toluene was not subtracted from the results for Toluene in the spiked samples.

The results across the scan for the low spike present a consistent pattern of recovery (Figure 3), except for the problem parameters noted above. The results across the scan for high spike are more variable (Figure 6). This suggests that this laboratory has more difficulty analyzing samples of a higher concentration. This may be due to curvature at the top end of their calibration range.

LABORATORY 1010

Results were not reported for the following parameters present in the spiking material: 1,2-Dichloroethane, Bromomethane, Chloromethane, trans-1,3-Dichloropropylene, and Vinyl Chloride. They used a 1% SP-1000 Carbopak gas chromatographic packed column. Schedule 3 of the MISA General Regulation (1) specifies the use of a capillary column. For regulatory purposes, this laboratory's results would not be acceptable. The results are reviewed for the purposes of improving laboratory performance.

The results for the low spike (VOL B) were under-recovered relative to the design value, for most parameters, except for Dichloromethane, cis-1,3-Dichloropropylene, and 1,1,2-Trichloroethane (see below for discussion of these three parameters). The results for the high spike (VOL C) had higher recoveries, ranging from 55-97% for many parameters. Some parameters appear to give problems, particularly Trichlorofluoromethane, 1,1-Dichloroethylene, and Carbon Tetrachloride.

The results for Tetrachloroethylene and 1,1,2,2-Tetrachloroethane were reported as less than ("<") a value. For the statistical calculations in Tables 1 and 2 (Appendix 1), and to calculate the percent recovery, half of the given value was used. The recovery for these two parameters is low relative to the design values, but this laboratory's results are semi-quantitative.

This laboratory reported that cis-1,2-Dichloropropylene, 1,1,2-Trichloroethane, and Dibromochloromethane co-eluted, and provided a combined result. One third of this value was entered into the spreadsheet for these parameters. These parameters are over-recovered relative to the design values, but this laboratory's results are semi-quantitative.

A result of 11.1 μ g/L for Dichloromethane was reported in the blank (VOL A). This is a common laboratory contaminant and may be a problem for this participant. The result for Dichloromethane reported in the blank was not subtracted from the results for the spiked samples. The result for Dichloromethane in the low spike gave a recovery of 119%, which is higher than the recovery for the other parameters in the scan, except for 1,1,2-Trichloroethane and cis-1,3-Dichloropropylene (see above). The recovery for Dichloromethane in the high spike does not differ from the majority of the other parameters in the same manner. This suggests that the background level of Dichloromethane in Laboratory 1010 affects low level samples but does not have the same effect on samples with higher levels of volatile organics.

Excluding the specific parameters mentioned above, reasonably consistent performance across the scan was demonstrated. There was no specific pattern of recovery based on the order of gas chromatographic elution. There are several specific problem areas that need to be addressed by this laboratory, as noted above.

Laboratory 1012

This laboratory did not report results for Dichlorobromomethane and trans-1,3-Dichloropropylene. They used a 1% SP-1000 60/80 Carbopack packed column, with a jet separator. Schedule 3 of the MISA General Regulation (1) specifies the use of a capillary column. For regulatory purposes, this laboratory's results would not be acceptable. The results are reviewed for the purposes of improving laboratory performance.

The results for the spiked samples (VOL B and VOL C) demonstrated variable recovery relative to the design values. This was a more noticeable problem with the low spike, as the recovery ranged from 0-110% (excluding Chloromethane and Dichloromethane; see below). For the high spike, the recovery ranged from 9-95% (excluding Chloromethane and Dichloromethane).

A value of 9.1 $\mu g/L$ was reported for Chloromethane. This is an unusual laboratory contaminant, due to the instability of this compound. This value was not subtracted from the spiked sample results, resulting in an apparently high recovery of this parameter in these samples. A value of 3.0 $\mu g/L$ was reported for Dichloromethane. This is a common laboratory contaminant and may have contributed to the higher recovery of this parameter from the spiked samples. This value was not subtracted from the spiked sample results.

The recovery across the scan does not demonstrate any pattern (Figure 1). Specific parameters appear to present problems for this laboratory, such as Chloromethane (see above), Trichloro-fluoromethane, 1,1-Dichloroethylene, Carbon Tetrachloride, and Tetrachloroethylene (low recovery of the latter four parameters). A similar pattern of performance was demonstrated in both spiked samples.

Laboratory 1013

This laboratory did not report results for 1,2-Dichloropropane, cis-1,3-Dichloropropylene, and trans-1,3-Dichloropropylene. They noted with their results that they could not explain the high result for Ethylene Dibromide in the high spike (VOL C), which they felt was out-of-line with the result from the other parameters. They used a SPB-5 60 m wide-bore, 0.32 mm I.D. capillary gas chromatographic column.

The results demonstrated good recovery relative to the design values, for most parameters, except for Ethylene Dibromide in the high spike. Carbon Tetrachloride and Tetrachloroethylene were under-recovered in both samples.

Nothing was reported in the blank sample (VOL A), so there is no evidence of any laboratory contamination problems.

Excluding the specific parameters mentioned above, reasonably consistent performance across the scan was demonstrated. There was no pattern of recovery based on the order of gas chromatographic elution. A similar pattern was demonstrated in both spiked samples.

Laboratory 1014

Laboratory 1014 included a note with their results indicating that they had had to repeat the analysis for Chloromethane and Bromomethane. The resulting headspace in the sample vials may have contributed to low results. After receiving the initial table of results, they reviewed their results for Dichloromethane and indicated that there was a background level of 9 μ g/L. They requested that this amount be subtracted from the initial results reported for VOL A, VOL B, and VOL C. The corrected values were used to calculate the percent recovery and were plotted in Figures 3 and 6. They used a J & W DB-624 30 m fused silica capillary column.

The results demonstrated variable recovery relative to the design value. This was more pronounced in the low spike (VOL B), with recoveries varying from 0-140%. Recoveries from the high spike (VOL C) varied from 40-143%.

Results for the blank sample (VOL A) were all reported as less than the MDL. For most parameters, the results suggest that there was no background contamination in this laboratory, other than the level of Dichloromethane, noted above.

The recovery across the scan appears to cluster the parameters into three groups. The majority of compounds are consistently recovered, with values between 80-110% of the design value. The second group of parameters were under-recovered: Chloroform, Carbon Tetrachloride, trans-1,3-Dichloropropylene, and Tetrachloroethylene. This group of parameters also shows a decreasing pattern of recovery within the over-all scan, based on the order of gas chromatographic elution. The third group of parameters were over-recovered: Benzene, 1,1,2-Trichloroethane, and Ethylene Dibromide. This laboratory has several specific problem areas to be addressed as noted above.

4.2 Oil and Grease Samples

OVERVIEW OF INTERLABORATORY PERFORMANCE - OIL AND GREASE

The results from all of the participants for the two spiked samples (O&G B and O&G C) were plotted using Youden's two-sample technique (2) in Figure 10. A line was drawn between the expected values and the origin to indicate the target area of precision.

The results demonstrate that, regardless of solvent used, individual laboratories demonstrate systematic bias when analyzing for Oil and Grease. While none of the participants demonstrated a high bias (relative to the design value), several laboratories demonstrate a low bias. The results are not affected by random error, as the majority of the points are very close to the line.

To further demonstrate that the between-laboratory variability is more significant than the within-laboratory variability, the F Test may be used. After converting to percent recovery, the between-laboratory variance may be estimated from the sums of the spiked samples (O&G B and O&G C). The within-laboratory variance may be estimated from the differences of the spiked samples. The calculations given in Table III demonstrate that the between-laboratory variability is more significant then the within-laboratory variability.

TABLE III - ESTIMATE OF BETWEEN-VS WITHIN-LABORATORY VARIABILITY

s_{Diff}^2	0.0063
S _{Sum}	0.1597
$F = \frac{s_{Sum}^2}{s_{Diff}^2}$	25.49

For $df_{Diff} = 11$, $df_{Sum} = 11$, and a 95% Confidence Interval, F = 2.81

The calculated F value is greater than 2.891, therefore the between-laboratory variability is significantly different from the within-laboratory variability.

There are several possible sources for the systematic error demonstrated by different participants. The method is sensitive to the timing of various steps in the analytical procedure (3). Variations in timing may cause different oils and greases to volatilize, resulting under-recovery. The purity of the solvent may affect the efficiency of the method. The temperature of the water bath also affects the recovery of the extracted materials.

The participants were not required to describe in detail their analytical procedure used for this interlaboratory study. It is desirable that the individual participants who have demonstrated systematic bias in this interlaboratory study, will strive to improve their performance. Future studies that include the analysis of Oil and Grease will attempt to monitor improvements in performance by the participants.

LABORATORY 1001

This laboratory used Freon for the extraction process. They had excellent recovery for both spiked samples (O&G B and O&G C), relative to the design values. However they appear to be biased high relative to the other participants who used the same solvent, both compared to the interlaboratory mean and median.

This laboratory also reported the highest level in the blank sample (O&G A). This suggests that there may be some background contamination in their laboratory. If the results for the spiked samples were corrected for the blank, the results would be closer to the interlaboratory mean and median.

The result for the effluent sample (O&G D) was also high, relative to the interlaboratory mean and median. This supports the above suggestion that there may be some background laboratory contamination.

LABORATORY 1002

This laboratory used Dichloromethane for the extraction process. They had excellent recovery for the spiked samples (O&G B and O&G C), relative to the design value. However they appear to be biased slightly high relative to the other participants, both for the interlaboratory mean and median.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was also high relative to the interlaboratory mean and median (excluding the result from Laboratory 1004; see Table 3, Appendix 1). As there was only one result for this sample from each participant, it is not possible to ascertain whether the high result is due to laboratory bias, or sample matrix effects.

LABORATORY 1003

This laboratory used Dichloromethane for the extraction process. They had good recovery of the low spike (O&G B) but under-recovered the high spike (O&G C). As a result, they are in good agreement with the interlaboratory mean and median for the low spike but appear to be biased low for the high spike. This laboratory may need to improve its within-laboratory precision. Participation in future interlaboratory studies will allow for assessing improvement their performance.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was low relative to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the low result is due to laboratory bias or the sample matrix.

LABORATORY 1004

Laboratory 1004 used Dichloromethane for the extraction process. Their results demonstrate good recovery relative to the design value for Sample O&G B and O&G C. They also demonstrate good agreement with the interlaboratory mean and median.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

A very high result for the effluent sample was reported by this laboratory. Compared to the results from all of the other participants, regardless of solvent used, this result appears to be an anomaly. While every effort was made to provide a homogeneous effluent to all of the participants, this laboratory may have received an aliquot that contained some additional extractable materials. Laboratory contamination may also have contributed to the anomalous result. The statistical calculations in Table 3, Appendix 1 were performed with Laboratory 1004's result deleted.

LABORATORY 1007

This laboratory used Freon for the extraction process. They had excellent recovery for the spiked samples (O&G B and O&G C), relative to the design value. However they appear to be biased slightly high relative to the other participants, both for the interlaboratory mean and median.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) showed good agreement with the interlaboratory mean and median.

LABORATORY 1008

This laboratory used Freon for the extraction process. They demonstrated consistent within-laboratory precision for the two spiked samples (O&G Band O&G C) but under-recovered relative to the design values. The results are also low relative to the interlaboratory mean. However this laboratory reported the median values from all of the participants who used Freon for the extraction. While this laboratory demonstrates comparable performance with many of the participating laboratories, some effort should be made to improve accuracy.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was low relative to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the low result is due to laboratory bias or the sample matrix.

LABORATORY 1009

This laboratory used Freon for the extraction process. They had good recovery of the low spike (O&G B) but under-recovered the high spike (O&G C). As a result, they are in good agreement with the interlaboratory mean and median for the low spike but appear to be biased low for the high spike. This laboratory may need to improve its within-laboratory precision. Participation in future interlaboratory studies will allow for assessing improvement their performance.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was low relative to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the low result is due to laboratory bias or the sample matrix.

LABORATORY 1010

This laboratory used Freon for the extraction process. They demonstrated good within-laboratory precision for the two spiked samples (O&G B and O&G C), but had very low recovery relative to the design values. Their results are also biased very low relative to the interlaboratory mean and median. These results suggest that this laboratory is consistent in their performance of the analysis for Oil and Grease, but need to improve their method to produce more accurate results.

This laboratory also reported a result for the blank sample (O&G A). If this value had been subtracted from the spiked sample results, their low bias would have been even more pronounced.

The result for the effluent sample (O&G D) was comparable to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the laboratory performed better when analyzing this sample as compared to their results for the spiked reagent water samples (O&G B and O&G C).

LABORATORY 1011

This laboratory used Freon for the extraction process. They under-recovered the low spike (O&G B) but had good recovery of the high spike (O&G C). As a result, they are in good agreement with the interlaboratory mean and median for the high spike but are biased low for the low spike. This laboratory may need to improve its within-laboratory precision. Participation in future interlaboratory studies will allow for assessing improvement their performance.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A). However the value represented as their lower detection limit (result was "<2 mg/L"), is higher than the MISA regulation MDL (for Group 25, MDL = $1000 \,\mu\text{g/L}$, Ref. 1). Some effort should be made to improve the lower detection limit.

The result for the effluent sample (O&G D) showed good agreement with the interlaboratory mean and median.

LABORATORY 1013

This laboratory used Dichloromethane for the extraction process. They had low recovery relative to the design values for the spiked reagent water samples (O&G B and O&G C). Their results also appear to be biased low relative to the interlaboratory mean and median. The within-laboratory precision could be also be improved. Participation in future interlaboratory studies will allow for assessing improvement their performance.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was comparable to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the laboratory performed better when analyzing this sample as compared to their results for the spiked reagent water samples (O&G B and O&G C).

LABORATORY 1014

This laboratory used Freon for the extraction process. They demonstrated good within-laboratory precision for the spiked reagent water samples (O&G B and O&G C). Their results are low relative to the design values. However they have good agreement with the interlaboratory mean and median. While this laboratory demonstrates comparable performance with many of the participating laboratories, some effort should be made to improve accuracy.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was high relative to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is not possible to ascertain whether the high result is due to any laboratory bias, or sample matrix effects.

LABORATORY 1015

This laboratory used Dichloromethane for the extraction process. They had excellent recovery for the spiked samples (O&G B and O&G C), relative to the design value. They have good agreement with the interlaboratory mean and median.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was lower than the interlaboratory mean, but had good agreement with the interlaboratory median.

5 REFERENCES

- ONTARIO REGULATION 695/88 as amended to Ontario Regulation 533/89 under the Environmental Protection Act; Effluent Monitoring - General
- Youden, W. J. and Steiner, E.H.; 1975; <u>Statistical Manual of the Association of Official Analytical Chemists</u>; Association of Official Analytical Chemists; ISBN 0-935584-15-3
- Standard Methods For the Examination of Water and Wastewater, 14th. Edition; 1976; American Public Health Association, American Water Works Association and The Water Pollution Control Federation; ISBN 0-87553-078-8

6 APPENDIX 1 - FULL DATA SET

Table 1	Volatiles, Samples VOL A, VOL B, and VOL C, Results in μg/L
Table 2	Volatiles, Samples VOL B and VOL C, Expressed as Percent Recovery of Design Value
Table 3	Oil and Grease, Samples O&G A, O&G B, O&G C, and O&G D, Results in mg/L
Table 4	Oil and Grease, Samples O&G B and O&G C, Results Expressed as Percent Recovery of Design Value
Figure 1	Volatiles, Labs 1001 and 1012
Figure 2	Volatiles, Sample VOL B, Labs 1002, 1003, and 1005
Figure 3	Volatiles, Sample VOL B, Labs 1007, 1009, 1013, and 1014
Figure 4	Volatiles, Lab 1010
Figure 5	Volatiles, Sample VOL C, Labs 1002, 1003, and 1005
Figure 6	Volatiles, Sample VOL C, Labs 1007, 1009, 1013, and 1014
Figure 7	Oil and Grease, Sample O&G B
Figure 8	Oil and Grease, Sample O&G C
Figure 9	Oil and Grease, Sample O&G D
Figure 10	Oil and Grease, Youden Plot of Samples O&G B and O&G C

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TR	0000		COLC	0700			COLC	OULC	OULC			300	0000	0 80	OULC	C	NOLC	ODEC	OUL C	COLC	2000	2000	0 0	0 0		VOLC	JOLE	SOLC	COLC

TEMBER SHORT BEINGER IN THE

PRINCED DE COLEMAN MEDITERRORGER DOME HERBE, CHR. HEBEL DE LEUR PERCHET PERCHETE MES L'HILPETT.
 LOCERATO PERCHETE DE CHE LUBEL.

THBLE 3 - INTERLABORATORY STUDY 89-1: OIL AND GREASE RESULTS EXPRESSED AS Mg/L (MISA GROUP 25)

015TRIBUTED: JANUARY 31, 1989 LABORATORY NUMBER SAMPLE DESIGN 1001 1002 1003 1004 1007 1008 1009	D: JAN ESIGN	1001	1, 198	1003	1004	LABORA 1007	TORY NE 1008	JMBER 1009	1010		1013	1011 1013 1014	1015	
08.5A 08.5C 08.6C	0 0 5	2523	5 5.5 21 20.3 12 18	11 4 11 7	11 18.5 7 112.5	रहु€	= 423	- 1 1 1 4 - 1 1 2 4	-1-9 -1-9	강토골시	1455 1455	7. 11.4 3.57 16. 12.4 13.07 7. 12.4 12.09	一切空四	
SOLVENT USED FOR EXTRACTION: F D LESEND: F - FREON; U - DICHLOR	ED TON: FRED	A 0 X	P PICHLI	D DROMETI	U BIFINE	-	F D U U F F F - C - C - C - C - C - C - C - C -	<u>-</u>	L	hain.	=	77	=	

z				
MEOIAR	0.5	9.6	14.5	~
МЯХ	2.0	5.2	21.0	12.3
MIN	0.0	2.2	4,1	3.8
STD DEV	0.7	1.1	4	5.5
MEAN S	9.7	3.8	15.6	7.6
1014	7.0	3,57	13, 92	12,28
1011		,	Ξ	>
1010	-	70 CJ	9,1	b. b.
1009	0.5	4.1	13.0	4.0
1008	0	3.6	14.5	3.8
1007	0.5	5.2	21	~
1001	N	n	121	12
FREON DESIGN	0	IJ	F	
SOLVENT: SAMPLE	D&GH	08.68	08/90	08860

SOLVENT:	DICHLOR	DMETHA	¥								
SAMPLE	DESIGN 1002	1002	1003	1004	1013	1015	MEAN S	STD DEV	MIN	MAX	MEDIAN
08GB	0	0.5	0.5	0.5	0	2.5	=	0.2	0	9.5	
0868	n	5.5	ব	4	4,5	27	4.0	2.7	4	50	
OREC	R2	20.3	11	18.5	12.3	Ξ	16,0	1.1	11	20.3	18
088D +		1.8	~	112.5	12.3	==	11.3	<u>-</u> З	~	10	

^{*} LABORRIORY 1004 RESULT EXCLUDED FROM STRITT-LICH CHECULTIONS.

NOTE: RESULTS MERE ELULIGED INTO THO DENOMES, LIFENNITHMS IN SOLVENT LISEL FOR EXTRACTION. STRITISTICAL DIRECTIONS, LINEATING THE SELF SECTION. 3.4 MEDISTICALS TO SELF SECTION.

THBLE 2 - INTERLABORATORY STUDY 89-1: VOLHTILE ORGANIUS (MISH GROUPS 16 & 17) RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE

DISTR	DISTRIBUTED: JANUARY 31, 1989	DESIGN			_	ABORATORY NUMBER	US YOU	MAFD									
SAMPL		(pdd)	1001	1002	1003	1005	1007	1009	1010	1012	1013	1014	MEAN ME	MERN MEDIAN MIN		MAX STI	STD DEV
VOLB	1, 1, 2, 2- RE FRACHLUPOR, LURINE	'n	40%	622	7.32	502	162	5963	7	1,652	2018	5.32	200	2017	2001	2 1	0.00
NOLB	1.1.2-1PICHLOROETHANE	W.	800%	1142	1.41127	111112	200	142	2010	2000	1000	1000	2000	200	100	7 11 17	4
NOLB	1, 1-DICHLOPOE THENE	LT.	1747	200	747	7.12	7.7	200	3 7 7	3 2 2	1200	10000	7007	777	N I	M 1	X 1
MUDITE	1. 1 DICHLORDE THYLENE	ŭ	2002	647	1.000	200	100	2000	200	3 2 2	7007	25.7	200	8.5	S.	174%	X ES
00 B	1 2-DICH GOODENZEME) L	3000	1 41.75		2014	000	200		70	1402	1362	13%	N. S.	Z	7 1 1 2 7	205
	1 3 Droin coormicant	n ı	202	145%	XZZO.	1012	200	252	N V	262	1002	116%	212	NI.	77. 4	1462	X55.
ALE SOLE	1, Z=UICHLURUE I HHME	ū	1967	1042 1042 1	100%	994	222	1122		200	1002	1062	1062	777	News	1,167	455
870FB	1,2-DICHLOROPPOPANE	S)	142%	1042	20,83	25.243	54%	2019	2025	7.07		200	80.7	NO.		140%	412
SOLB	1, 3-DICHLROBENZENE	u)	88%	1362	80.02	972	582	BRZ	27.15	722	1402	1002	212	1000	1 1	1400	21.5
COLB	1,4-DICHLOROBENZEME	IJ	362	128%	20,84	266	U22	298	412	822	100%	11.2%	717	40	412	7,007	2000
COLB	BROMOFORM	ທ	72%	1022	2001	2101	4 22	88%	N. C.	7.2%	1002	1107	i N	1147	2000	110%	3 7 6 7
B 700.	BEDMOMETHRNE	រោ	2002	1162	2011	11312	20	5202		1102	1402	E	1222	1102	200	200.54	1.000
a non	CHRBON TETERCHLORIDE	เก	1022	48%	222	222	0%	222	202	162	80%	72%	472	N	1 2	1102	2000
NOLB NOLB	CHLOROBENZENE	D)	0. 7.4.0	100%	25.62	83%	382	80%	34%	642	80%	1002	70%	200	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	23,001	262
AUC.	CHLUKUFUKA	ហ	142%	100%	787	672	102	68%	402	622	80%	74%	72%	717	107	140%	7.02
NOLB	CHLOROMETHRNE	ល	820%	174%	1122	269	20	118%		2502	10002	8.22	1992	11.00	6	10.0	2462
NOCE.	cis-1,3-DICHLOROPROPYLENE	ហ	120%	64%	1042	1162	202	362	2162	1062		1062	286	1062	25	7187	567
BOO	DICHLOROBROMOME THANK	n	1462	104%	1022	268	52%	382	4.2%		1002	1162	94%	1002	20.10	1467	N I
OULB	ETHYLENE DIBRUMIDE	ın	72%	124%	1367	1022	462	1102	X8.7	206	120%	1762	1022	TORK	20.00	1267	417
SOLB SOLB	DICHLOROMETHRNE	Ŋ	522	136%	762	212	70	42%	1192	1762	200	1002	1042	8052	8	2 E E	292
BOOK	IE IPHCHLURUE I HYLENE	n	402	64%	1142	37%	40%	202	172	20.50	80%	66%	522	A55.2	17%	1142	74%
BOOK	trans-1,2-DICHLORUEIMYLENE	IO I	260%	120%	1002	67%	20	762	542	862	80%	1762	1042	X/B	20	2092	712
HOLE	trans-1,3-DICHLOROPROPYLENE	រោ	40%	88%	762	62%	8	2U9				662	49%	F.22	DZ	KBR	NO.
B 000	TRICHLURUE I HYLENE	ו מו	1462	122%	200	103%	82	762	41%	642	208	144%	872	842	W.	1462	777
0000	INTERCRINE LUCKIONE I HANE	D I		34%	602	36%	8	202	X07	20	1002	1142	46%	362	022	1.42	402
	OTHER CHUMIUE	וכו	640X	1187	112%	28.9	20	M2K		1022	200	1442	1492	1022	20	1,40%	1892
0000	DELICENE	n i	154%	242	7.47	7.07	2 2	842	20,100	86%	1002	17.2%	97%	24%	302	1722	412
	ETHYLBENZENE 301 11010	ı,	647	1133	200	10002	SPS	242	40%	208	1002	116%	85%	2117	A02	1162	2000
AUCE COLE	TULUENE Star foot	ומ	28%	104%	2002	26.8	74%	662	412	722	209	146%	812	132	412	1462	402
A OLE	O-AYLENE XX DAY	ומו	80%	1162	9EZ	212	1102	1042		787	7001	120Z	286	21111	2009	707	192
000	STANTENED OF STANT	ח נו	803						7 7	687			572	14,022	4 322	1182	1.3%
NO B	THE SAME ONE TO	0 0	2002		-					787							
	OF BMD DEXTEND	20		262	27.50	1972 1972	46%	X95.			205	1042	812	HVZ	462	HMZ	212
	o mile protestas	0.1							4132								

		TABLE 2		- INTERLABORATORY STUDY 89-1: VOLNTILE ORGANICS (MISA BROUPS 16 \aleph RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE	SHTORY XPRESSI	STUDY ED AS	89-1: PERCEN	VOLAŤ T RECO	TLE OR VERY D	GANICS F DESI	CMISH GN VHL	GROUP	S 16 8	170			
OISTR	OISTRIBUTED: JANUARY 31, 1989 SAMPLE PARAMETER	(ppb)	1001	1002	1003	.нВОКНТ 1005	LHBORHTORY NUMBER 1005 1007 1009	MBER 1009	1010	1012	1013	1014	MEAN P	MEAN MEDIAN MIN	MIM	MHX v	STD D
COLC	1, 1, 2, 2- 1E FEPURICHURY THENE	30		8	<u>N</u>	8.7	40%	.11%	22	12120	20.44	2	1,72	-	21.5	7	
COET	1, 1, 2 - TP TCHLUPOETHENF	08		11.3	HAIZ	7.92	219	122	20 11 15	200	FIRE	1.42	144%	100	13.00	100	-
OULC	1, 1 - O LCPIL OP/OF THENE	8		2002	717	1.622	20	202	200	4.12	2000	200	2544	77	24	2	
NOLC	1,1-DICHLOPUETHYLENE	9		20174	22.52	46%	717	2546	200	TEX	2004	HIX	4,12	22	112	HIZ	
NOLC	1,2 DICHLOPORENZENE	S		1.41%	20	2901	7.4%	777	2010	707	2007	10802	Z E	20-11	2002	7 1	
NOLC	1,2 DICHLOPUE PHRNE	8		1162	27111	27.01	212	B3%		260	14022	1 3502	75 F	20.00	212	1 1922	
ODFC	1,2 DICHLORNPRUPRNE	200		27.71	2015	17.4%	20164	58%	2.24	2002		1002	6772	7112	112	10.20	
2707	1, 3-DICHLPUBENZENE	8		11.2	77.0%	25	717	672	27.74	200	1,352	1012	862	7.7	25.50	1.452	
0000	1,4-DICHLUROBENZENE	02		1142	0.4%	11.0%	2.9%	672	74%	25.53	200	1102	862	2002	25,52	11.92	
2700	BROMOFORM	50		11.22	1112	142	224 *4	1.22	5552	652	11552	1152	206	25,434	1.3%	1152	_
0700	BPOMOMETHBNE	30		11862	110%	200	4,320	3172		208	25.95	42%	1062	1182	4.3%	21.72	0
SOFC	CARBON TETRRICHLORIDE	50		44%	46%	200	1.3%	29%	217	182	. 652	552	24.2	35.22	1.32	252	-
NOLC	CHLOROBENZENE	20		2002	0.0%	N Z	2002	45%	27.2	200	70%	1987	67%	512	452	206	_
NOLC	CHLOROFORM	50		N IS	702	202	2442	422	707	208	787	682	63%	5000	34%	215	T
NOLC	CHLOROME THANE	8		15.3%	11142	85,	262	55%		1102	MAN	712	86%	812	23%	11.63%	77
0000	cis-1,3-DICHLOROPROPYLENE	50		ZP2	1132	75%	71%	7.3% 7.3%	2269	8.5		133%	131%	862	ZE	5372	151
ODDC:	DICHLOROBROMOMETHRNE	20		10.72	1062	89%	612	88%	85%		582	1262	88%	952	6.1%	1.36%	n)
3700	ETHYLENE DIBROMIDE	2		1202	140%	04%	202	93%	942	45%	FULLS	1432	1602	582	269	SUUS	18
COLC	UICHCOROME INTER	Z		1062	74%	7.47	N	2337	200	1007	1002	1052	80%	296	20	1502	Ť
0000	TETRACHLOPOETHYLENE	50		25.5	1142	222	252	282	26%	1252	402	40%	43%	292	233	114%	
0000	trans-1,2-DICHLOROETHYLENE	20		1112	20%	X 200	1.9%	52%	288	452	202	112%	672	592	13%	112%	9
2000	trans-1, 3-U1CHLORUPROPYLENE	200		Mez	31%	298	202	49%				259	552	6.4%	20	212	.70
OULC.	IRICHLURUE I HYLENE	2		1102	04%	26%	41%	42%	542	2822	202	206	702	E-472	41%	1102	7.0
2000	TRICHLUPUPLDURUMETHANE	25		22	4.2%	262	162	26%	242	22.5	202	253	200	262	92	282	1
COLL	VINYL CHUUMIUE	3		11.2%	N T T	36%	212	362		2 5	B552	242	269	SBX	30%	1122	
2000	BENZENE	200		1012	N N	242	222	492	202	2013	HEN	2000	84%	85%	492	1 JBZ	.73
0 TOS	ETHYLBENZENE	5		1052	CRIZ	76%	18%	262	6.3%	298	2002	95%	269	ZUZ	18%	1115%	-
2700	TOLOLOPIA	20		1052	1022	29%	2562	35%	2002	65%	2005	2000	75%	1,52	35%	1072	- 7
2700	O-XYLENE	5		114%	272	1162	5.3%	69%		P-0.22	852	1002	2002	912	602	1162	0)
ODEC ODEC	m-XYLENE p-XYLENE	88							5,12	202							
NOFC	m- AND p-XYLENE	40		54%	HBK	209	12%	202		5	702	852	2562	50.150	1.2%	X	.5.
2700	o- AND p-XYLENE	40							55.52								

THBLE 4 - INTERLABORATORY STUDY 89-1: OIL AND GREASE (MISA GROUP 25) RESULTS EXPRESSSED AS PERCENT RECOVERY OF DESIGN WALUE

DISTRIBUTED: JANUARY 31, 1989

ā	252 262 263
QI QU	j
ò	
7	44Z 46Z
i d	
CTD	50.50
NG LIN	
4101	212
101	•
0101	44%
IMBER 1009	#102 BEIZ
HTORY NUMBER	727
.AB0RA1	104Z 105Z
1001	1007
FREON	og
SOLVENT:	04.6B 0x.6C

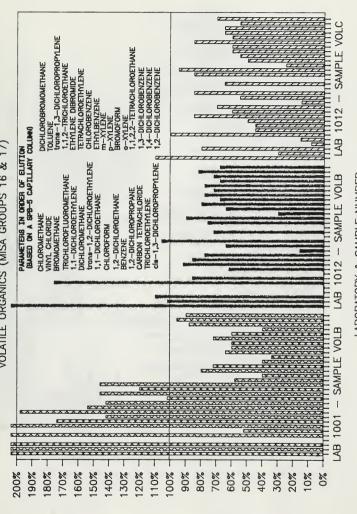
282 982 DESIGN 1002 1003 1004 1013 1015 MERN STD DEV MIN MRX MEDIRN 1110Z 1112Z 48X 25X 252 B02 1002 1002 48% 6.2% 997 937 807 557 SOLVENT: DICHLOROMETHRNE 110X 102X s S SHMPLE 08 GB 08.60

NOTE: RESULTS MERE FLUVIDED INTO THO GROUPS, FLEPENDINS ON SOLVENT USED FUR EXTRACTION. RBOVE STRITISTICAL CALCULLITURS GONE ON SEPPIRATE DATH SETS DALLY.



INTERLABORATORY STUDY 89-1 FIGURE

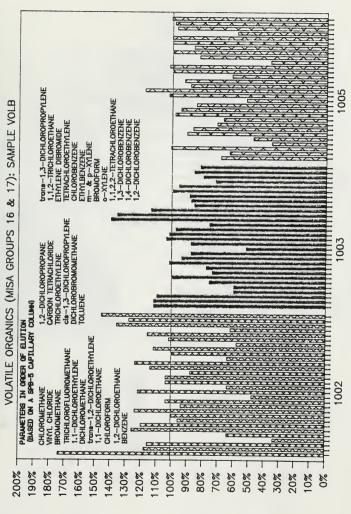
VOLATILE ORGANICS (MISA GROUPS 16 & 17)



LABORATORY & SAMPLE NUMBER



FIGURE 2 - INTERLABORATORY STUDY 89-1

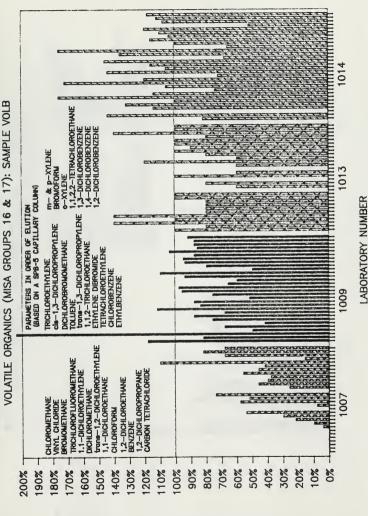


LABORATORY NUMBER

PERCENT RECOVERY OF DESIGN VALUE

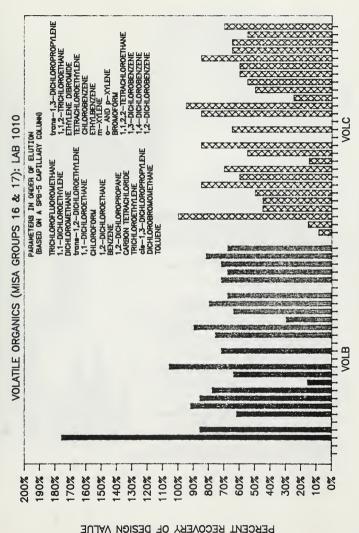


INTERLABORATORY STUDY 89-1 FIGURE 3





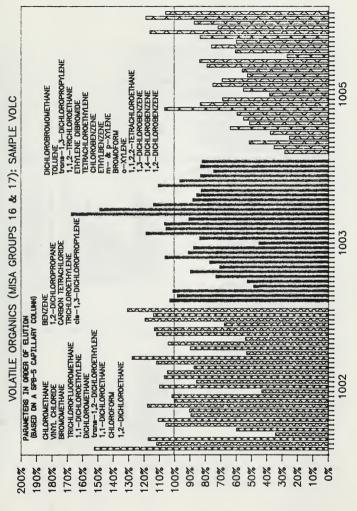
INTERLABORATORY STUDY 89-1 FIGURE 4 -



SAMPLE NUMBER



INTERLABORATORY STUDY 89-1 FIGURE 5



LABORATORY NUMBER



INTERLABORATORY STUDY 89-1

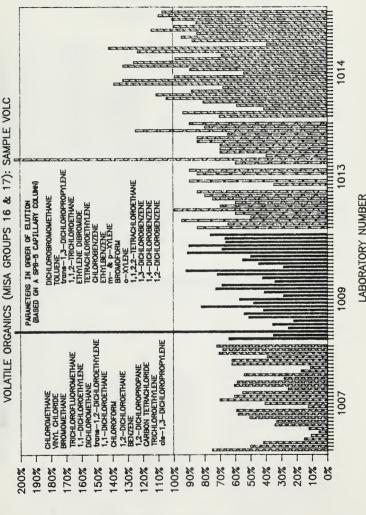




FIGURE 7 - INTERLABORATORY STUDY 89-1

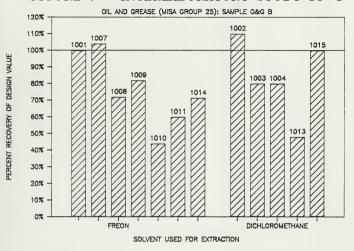


FIGURE 8 - INTERLABORATORY STUDY 89-1

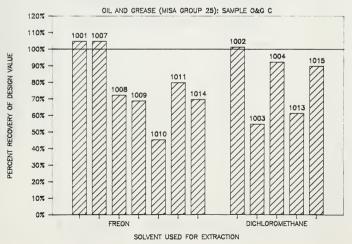
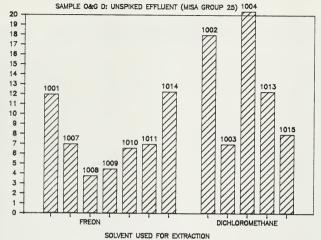


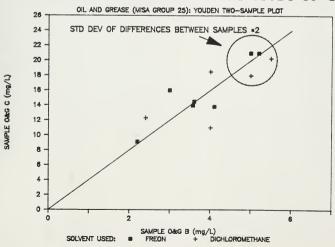


FIGURE 9 - INTERLABORATORY STUDY 89-1



RESULTS IN mg/L

FIGURE 10 - INTERLABORATORY STUDY 89-1





7 APPENDIX 2 - LIST OF PARTICIPANTS AND CORRESPONDENCE

LIST OF PARTICIPANTS

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Contact: Dr. John Leach

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF TRACE ORGANIC COMPOUNDS AND SOLVENT EXTRACTABLES STUDY NO. 89-1

INTRODUCTION

Private laboratories receiving this notification are invited by the Ontario Ministry of the Environment to participate in an interlaboratory variability study of spiked reagent water conducted using MISA analysis protocols. Laboratories interested in participating in this program, scheduled for the week of January 30, 1989, should contact Sylvia Cussion at (416) 235-5842 or Catherine Doehler (416) 235-6055 of the Ministry of the Environment to confirm participation no later than January 25, 1989. All participants should follow-up telephone acceptance with written confirmation (FAX - (416) 235-5744) by January 25, 1989.

BACKGROUND

This study is being conducted to assist laboratories in assessing their analytical performance. All procedures should follow those principles and protocols outlined in the MISA regulations (Ontario Reg. 358/88). Sample sets will include three samples per scan: a blank, a low spike (approx. 5 times the MDL) and a high spike (approx. 20 times the MDL). Sample sets for Oil & Grease (MISA Group 25-Solvent Extractables) will include 4 samples: a blank, low spike, high spike, and a "natural" matrix sample.

NOTE: Any laboratory that does not have a copy of the MISA general regulations should contact Catherine Doehler for additional information.

The following scans are to be included in this round robin:

<u>Volatiles</u> (to be analyzed by GC/MS <u>and/or</u> GC/ECD/FID) <u>MISA Groups 16 & 17</u>

Time Limit: 7 days storage

1,1,2,2-Tetrachloroethane 1,1,2-Trichloroethane

1,1-Dichloroethane 1,1-Dichloroethylene 1.2-Dichlorobenzene

1,2-Dichloroethane 1,2-Dichloropropane

1,3-Dichlorobenzene 1,4-Dichlorobenzene

1,4-Dichlorobenzene Bromoform

Bromomethane

Carbon tetrachloride Chlorobenzene Chloroform

Chloromethane cis-1,3-Dichloropropylene

Dibromochloromethane Ethylene dibromide

Dichloromethane (Methylene Chloride)

Tetrachloroethylene

trans 1,2-Dichloroethylene trans 1,3-Dichloropropylene

Trichloroethylene

Trichlorofluoromethane

Vinyl chloride (Chloroethylene)

Benzene Ethylbenzene Toluene

o-Xylene m-Xylene

p-Xylene

Solvent Extractables (liquid/liquid extraction) MISA Group 25

Time Limit: 7 days storage

Oil & Grease

SCHEDULE

During the week of January 30, 1989 participating laboratories will receive a total of seven (7) samples for analysis. Three (3) samples will be for the analysis of Volatiles and four (4) will be for the analysis of Solvent Extractables. All samples will be spiked reagent water samples, with the exception of Solvent Extractables, which will include a fourth unspiked sample.

Participating laboratories are expected to analyze the samples within the time limits specified in Schedule 2 of the general MISA regulations (Ontario Reg. 358/88). Blank reporting forms will be provided with the samples. Results for all analyses are to be reported withinty (30) days of receipt of the samples to Sylvia Cussion/Catherine Doehler at the following address:

Ministry of the Environment Laboratory Services Branch Laboratory Computer Systems - QA/QC Section 125 Resources Rd., P.O. Box 213 Rexdale, Ontario M9W 5L1

SUMMARY OF RESULTS

All participating laboratories will be assigned a unique identification code. All laboratories will receive a complete set of the results, including a ranking for each laboratory where they will be identified only by their identification code. Recommendations made by the MOE will also be provided to the individual labs. Results will remain confidential and will only be released with the written permission of the individual participants.

It is the intent of this round robin (along with others) to assess the interlaboratory variability and detection capability for a broad range of organics and inorganics.

Ontario Ministry of the Environment Laboratory Services Branch LCS-QA/QC Section 125 Resources Rd. Rexdale, Ontario M9W 5L1 (416) 235-5842 or 235-6055 FAX (416) 235-5744

January 31, 1989.

TO: PARTICIPANTS OF MOE ROUND ROBIN 89-1

Please find enclosed four (4) 1000 mL amber bottles and three (3) 40 mL clear bottles. The samples are labelled as follows:

1000 mL Amber Bottles	40 mL Clear Bottles
O&G A	VOL A
O&G B	VOL B
O&G C	VOL C
O&G D	

If you are missing any of the above items, please contact us at the above phone number immediately.

Your participation in MOE Round Robin 89-1 is greatly appreciated by the Laboratory Services Branch of the Ministry of the Environment.

As was stated in the notification distributed January 18, 1989, samples should be analyzed using the principles and protocols outlined in the MISA general regulation (Ontario Reg. 358/88). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limits for storage were indicated in the advance notification. Results are to be reported within thirty (30) days of reception of the samples. Report forms are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact us if there are any problems or questions re the round robin.

Your lab identification number is:

Sincerely,

Sylvia Cussion Lab Quality Audit Scientist (416) 235-5842 Catherine Doehler MISA Audit Scientist (416) 235-6055 Laboratory Services Branch LCS-QA/QC Section 125 Resources Rd. Rexdale, Ontario M9W 5L1 (416) 235-5842

June 14, 1989

TO: PARTICIPANTS OF MOE LINTERLAB VARIABILITY ROUND ROBIN 89-1

Thank you for your participation in the Interlaboratory Variability Study 89-1 conducted in January 1989. Your efforts are greatly appreciated by the Ministry.

Attached are all the results reported to me as of the end of March 1989. Not all the original participants were able to report results. Listed for each sample and parameter are the expected values, the results reported by each participant, the mean, maximum result, minimum result, and the standard deviation. For labs that reported a result for a particular parameter in Groups 16 & 17 for Samples B and C using a < or ND (not detected) designation, the value was entered as 0. In group 25, all < values were entered 1/2(<) for calculation purposes. All participants are designated by their identification code only.

A formal report is presently being written, but I regret that it will not be completed until July 1989. All participants will receive a copy as soon as it becomes available.

Please contact me if you have any further questions.

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist\

Attachment





